

Alkaline treatment of high-solids sludge and its application to anaerobic digestion

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ABSTRACT

High-solids anaerobic digestion is a promising new process for sludge reduction and bioenergy recovery, requiring smaller digestion tanks and less energy for heating, but a longer digestion time, than traditional low-solids anaerobic digestion. To accelerate this process, alkaline sludge disintegration was tested as a pretreatment method for anaerobic digestion of high-solids sludge. The results showed that alkaline treatment effectively disintegrated both low-solids sludge and high-solids sludge, and treatment duration of 30 min was the most efficient. The relation between sludge disintegration degree and NaOH dose can be described by a transmutative power function model. At NaOH dose lower than 0.2 mol/L, sludge disintegration degree remained virtually unchanged when sludge total solids (TS) content increased from 2.0 to 11.0%, and decreased only slightly when sludge TS increased to 14.2%. Although high-solids sludge required a slightly higher molarity of NaOH to reach the same disintegration level of low-solids sludge, the required mass of NaOH actually decreased due to sludge thickening. From the view of NaOH consumption, sludge TS of 8–12% and a NaOH dose of 0.05 mol/L were optimum conditions for alkaline pretreatment, which resulted in a slight increase in accumulative biogas yield, but a decrease by 24–29% in digestion time during the subsequent anaerobic digestion.

Key words | anaerobic digestion, biogas, pretreatment, sludge

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INTRODUCTION

The amount of sewage sludge discharged as a byproduct from wastewater treatment plants (WWTPs) has been increasing rapidly due to enlarged wastewater treatment capability and more stringent effluent regulations. To reduce the amount of sewage sludge requiring disposal, anaerobic digestion (AD) is widely used around the world as a sludge treatment process. This process can also stabilize sludge, transform organic matter into biogas and limit possible odor problems associated with residual putrescible matter (Appels *et al.* 2008). The capability to recover energy from waste biomass, such as sewage sludge, enhances the viability of AD as a sludge treatment technique.

Conventional anaerobic digestion (CAD) commonly treats sludge with a total solids (TS) content of 2–5%, because mixing, heat transfer and pumping all become inefficient and expensive as a result of high viscosity of sludge with greater TS (Jolis 2008). However, AD of low-solids sludge is limited in small-scale WWTPs due to economic limitations and inadequate planning (Duan *et al.* 2012).

Moreover, there is commonly not enough space for traditional big digesters in many small-scale WWTPs, especially in highly urbanized areas. In general, most of these WWTPs have a system of sludge dewatering, and the dewatered sludge with water content of about 80% is discharged and transported for further treatment or disposal. For the dewatered sludge, high-solids AD would be an attractive option for reducing sludge volume and recovering bioenergy. This process can treat sludge with 6–20% TS and has been recognized to be advantageous over traditional low-solids AD due to its requirement of smaller digesters and less energy for heating (Guendouz *et al.* 2008).

During sludge AD, hydrolysis of sludge particles is the rate-limiting step (Eastman & Ferguson 1981). Thus, CAD requires a long sludge retention time (SRT) of about 20–30 days (Weemaes & Verstraete 1998). When sludge concentration increases, SRT increases such that AD of high-solids sludge is a relatively slow process compared to CAD. To improve digestion efficiency, the common logical

approach is to break down sludge particles and microbial cells prior to digestion (Pavlostathis & Gossett 1986). Many sludge disintegration technologies, including alkaline treatment, have been used as pretreatment methods prior to AD. Alkaline treatment can disrupt sludge flocs and cells, release inner organic matter and accelerate sludge hydrolysis, and consequently improve the performance of subsequent AD (Lin *et al.* 1997; Kim *et al.* 2003; Cassini *et al.* 2006). Extracellular polymer substances (EPS) typically hold sludge particles together to form flocs. However, high pH causes proteins to lose their natural shapes and prompts saponification of lipids and hydrolysis of RNA. Strong alkali conditions solubilize organic gels because of not only chemical degradation but also ionization of hydroxyl groups, leading to extensive swelling and subsequent solubilization (Neyens *et al.* 2004). After destruction of EPS and gels, cells are exposed to a high pH environment in which they cannot keep the appropriate turgor pressure. The loss of turgor pressure, coupled with saponification of lipids, causes the destruction of cells and the release of the cellular contents (Li *et al.* 2008).

During alkaline sludge treatment, alkali type and dose, treatment duration and sludge solids concentration are the main factors impacting sludge disintegration. The preferred reagent, in most cases, has been sodium hydroxide (NaOH), which yields greater solubilization efficiency than calcium hydroxide [Ca(OH)₂] (López Torres & Espinosa Lloréns 2008). The effects of NaOH dose, treatment duration and sludge concentration on the effectiveness of alkaline treatment have been evaluated only in the range of sludge TS from 2 to 5%, which is also the range commonly used in CAD (Appels 2008). Considering sludge disintegration efficiency, NaOH dose usually has been controlled at a level of 0.02–0.1 mol/L, with an optimized treatment duration of 30 min (Lin *et al.* 1997; Navia *et al.* 2002; Li *et al.* 2008; Li *et al.* 2012). Alkaline treatment and the subsequent neutralization of the residual alkali bring a certain concentration of sodium ions into the sludge, which possibly results in inhibition of anaerobic bacteria. In general, 3.5–5 g/L sodium ions moderately inhibits the activity of mesophilic methanogens, and 8 g/L sodium ions leads to strong inhibition (McCarty *et al.* 1964). Correspondingly, the NaOH dose should be lower than 0.15–0.22 mol/L. When sludge TS increased in the range of 2–5%, the dissolved organic matter per unit sludge solids had almost no change under the same pH (Xiao & Liu 2006); this result suggests that the same level of sludge disintegration can be achieved using less alkali when sludge is thickened to increase its solids concentration. Thus, high-solids sludge is an attractive object for both alkaline treatment and AD.

Alkaline treatment of low-solids sludge has been widely investigated and verified to be effective for dissolving sludge organic matter and improving the subsequent AD (Lin *et al.* 1997; Navia *et al.* 2002; López Torres & Espinosa Lloréns 2008; Li *et al.* 2012). However, in these previous investigations, sludge solids contents were all <6%. So far no reports have focused on alkaline treatment of high-solids sludge and its impact on the subsequent high-solids AD. Thus, this study was designed to accelerate AD of high-solids sludge through alkaline pretreatment. At first, alkaline sludge treatment was investigated in a wide sludge TS range from 2 to 15%, and the optimized conditions of alkaline sludge pretreatment were determined. After that, batch AD experiments were carried out in order to verify the effect of alkaline pretreatment on AD of high-solids sludge.

MATERIALS AND METHODS

Sludge samples

With an increasing interest in AD, centralized digestion facilities were built outside of WWTPs in some Chinese cities. Hence, dewatered sludge was first transported from WWTPs to digesters at other sites, and then diluted as feedstock for AD. Thus, sludge samples used in this study were dewatered sludge diluted by deionized water. The dewatered sludge was collected from a local full-scale municipal WWTP and stored at 4 °C until it was used in experiments. In this plant, a biological aerated filter process was applied to clean the wastewater. The discharged sludge, composed mainly of primary sludge with a small amount of biofilm sludge, was conditioned with polyacrylamide and dewatered to a water content of about 80% by centrifugation. The collected dewatered sludge was diluted to samples with differing TS concentrations ranging from 2.0 to 4.2%. The volatile solids (VS) were 48–50% of TS. Before alkaline treatment, total chemical oxygen demand (TCOD) of these samples was calculated by their VS and chemical oxygen demand (COD) per unit VS (COD/VS, g/g) and were measured to be 1.12 ± 0.11 g/g. The initial soluble chemical oxygen demand (SCOD₀) of untreated samples ranged from 1,246 to 4,107 mg/L.

Alkaline sludge treatment

Alkaline sludge treatment was conducted in 2.0 L batch mixed reactors. After NaOH was added, the sludge samples were stirred at 240 rpm for 30, 60, 120 and 240 min,

respectively. The solubilization of sludge organic substances was measured using soluble chemical oxygen demand (SCOD). Every test was carried out four times. The average value was used to evaluate sludge disintegration degree (DD_{COD}), which was defined as the ratio of the increase in SCOD due to alkaline treatment to the maximum possible SCOD increase

$$DD_{\text{COD}} = \frac{\text{SCOD} - \text{SCOD}_0}{\text{TCOD} - \text{SCOD}_0} \quad (1)$$

Biochemical methane potential tests

Biochemical methane potential (BMP) tests were carried out to investigate the influence of alkaline pretreatment on AD of high-solids sludge. The diluted dewatered sludge (the substrate) was first disintegrated with NaOH and then adjusted to pH 7–8 with HCl. The inoculum was the concentrated digested sludge discharged from a laboratory-scale semi-continuous mesophilic digester with an SRT of 20 days. The substrate and the inoculum were mixed according to the ratios shown in Table 1. After the mixture was added into a 250 mL jar, the jar was flushed with nitrogen in order to remove oxygen and then sealed with a rubber plug. The jars were placed in a shaking water bath (SHA-C, Zhongda Instrument, Jintan City, China) to maintain the reaction temperature at $35 \pm 2^\circ\text{C}$. A close-fitting polyvinyl chloride connection tube was inserted into a hole pierced through the center of each jar plug for transferring the

biogas to the subsequent measurement device. The biogas yield was recorded by displacement of a saturated sodium chloride solution until there was no more biogas production. A 'blank' test was set in order to determine the biogas yield of the inoculum itself, so that it could be subtracted from the measured biogas yield, leaving the yield from only the treated sludge. Every condition was repeated twice, and the average was used for comparisons.

Measurement of sludge viscosity

Sludge viscosities were measured using a rate-controlled viscometer (SNB-2, Nirun, Shanghai) at ambient temperature (21°C). Before measurement, sludge samples were stirred gently for a short time to ensure the samples were homogeneous, yet retained the essential structure of particle-to-particle bonds that existed. Four hundred millilitres of prepared sample were first put into a 400 mL beaker (with the body diameter of 82 mm), into which the spindle of the viscometer was immersed to a depth indicated by the manufacturer (just below the liquid level). The measuring speed (the spindle speed) was set as 40–80 rpm. The measuring time was set as 30 s; the short test time was selected to avoid inaccurate results that could be derived from the sedimentation of some particles from the sludge suspension.

Analytical procedures

TS, VS, pH and COD were determined according to standard methods (MEP 2002). When measuring SCOD, sludge samples were centrifuged at 5,000 g for 10 min, and the supernatant was filtered through a membrane with a mesh size of $0.45\ \mu\text{m}$. The filtrate was used to determine the SCOD.

Table 1 | Arrangement of biochemical methane potential tests

Parameters	G0	G1	G2	G3
Substrate				
Mass (g)	100.0	100.0	100.0	100.0
TS (%)	8.0	8.0	8.0	8.0
VS (%)	48.4	48.4	48.4	48.4
NaOH dose (mol/L)	0	0.05	0.10	0.15
Inoculum				
Mass (g)	100.0	100.0	100.0	100.0
TS (%)	8.0	8.0	8.0	8.0
VS (%)	37.0	37.0	37.0	37.0
Mixture				
Mass (g)	200.0	200.0	200.0	200.0
TS (%)	8.0	8.0	8.0	8.0
RI/S ^a	0.76	0.76	0.76	0.76

^aRI/S: the ratio of inoculum VS to substrate VS.

RESULTS AND DISCUSSION

Effects of alkaline sludge treatment

During alkaline sludge treatment, the effect of sludge disintegration was represented by the variation of SCOD (Figure 1). DD_{COD} increased directly with NaOH dose and treatment duration, whether for high-solids sludge or for low-solids sludge. The most efficient treatment duration was 30 min, as the further increase of DD_{COD} was very limited when treatment duration extended from 30 to 60, 120 or 240 min. For the sludge with 2% TS, the released COD in

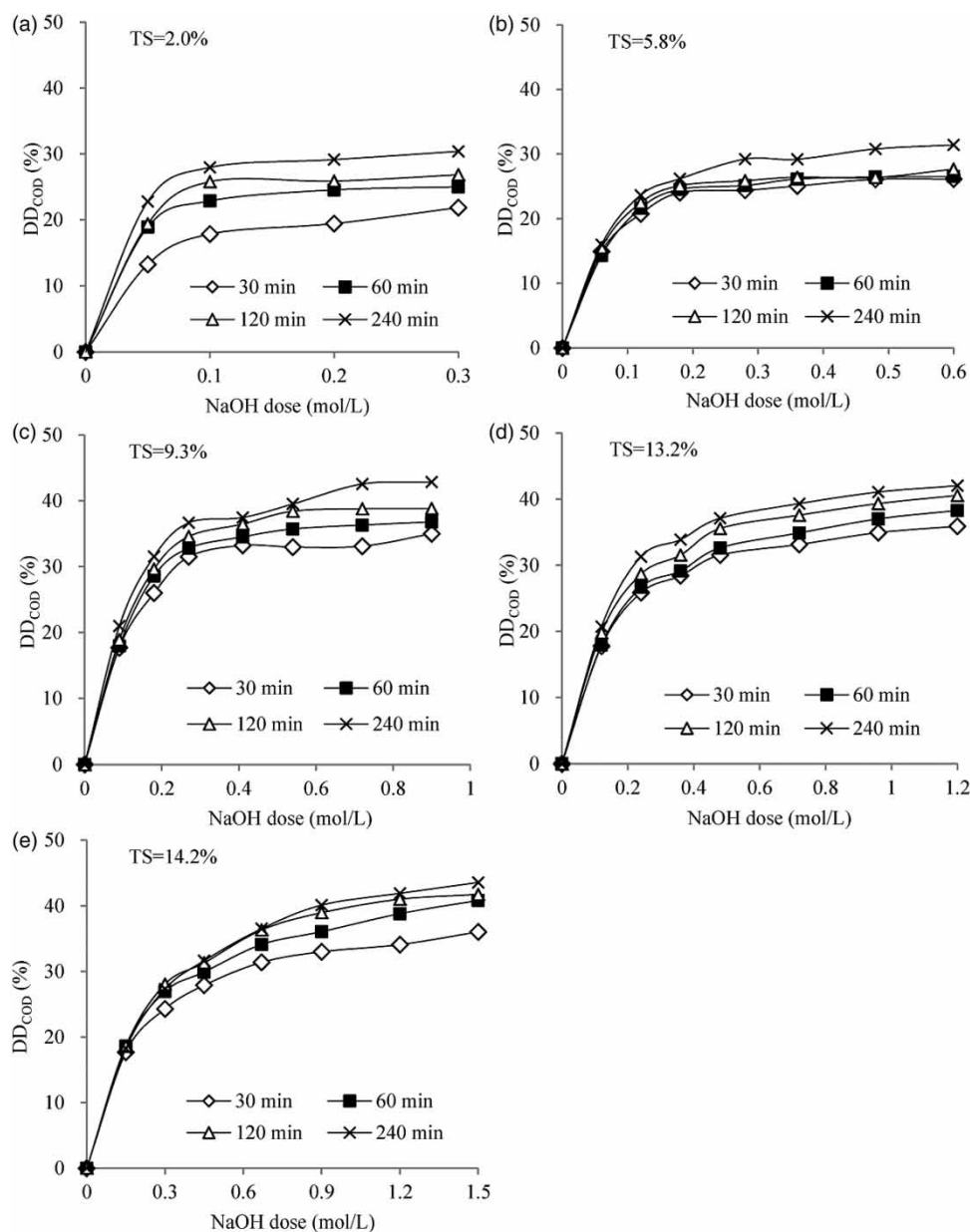


Figure 1 | Influence of NaOH dose, treatment duration and sludge TS on sludge DD_{COD} .

the first 30 min accounted for 60–72% of the total released COD in 240 min. It was previously reported that the increase of sludge SCOD can be divided into two stages: an initial rapid stage of 30 min and a subsequent slow stage (Li *et al.* 2008). In the first 30 min, the solubilization quantity was 60–71% of total solubilized organic matter in 24 h. The treatment duration of 30 min was also selected in other works (Navia *et al.* 2002; Cai *et al.* 2004; Xiao & Liu 2006). In this study, it was verified that the treatment duration of 30 min was the most efficient for alkaline treatment of high-solids sludge with TS of 5.8–14.2%.

NaOH dose was another factor influencing sludge disintegration. More organic matter was dissolved with higher NaOH doses, but the increment of dissolved organic matter reduced gradually at each successive dose. From the view of alkaline efficiency, 0.1 mol/L NaOH was appropriate for the disintegration of sludge with 2.0% TS, which was also the optimum dose found in previous work (Li *et al.* 2012). For high-solids sludge with 9.3–14.2% TS, the most efficient doses were found to be in the higher range of 0.2–0.3 mol/L. However, at the same dose of 0.1 mol/L, sludge DD_{COD} was almost constant even when

sludge TS increased from 2.0 to 11.0% (Figure 2). This showed that the sludge disintegration effect was relatively unaffected by sludge concentration in this TS range. In other words, sludge disintegration levels were mainly determined by NaOH molarity. This discovery was in accordance with a previous report on alkaline treatment of low-solids sludge with TS of 2–4% (Xiao & Liu 2006). According to NaOH molar concentration and sludge solid concentration, NaOH dose on the basis of mass ratio (g/g TS) can be calculated. At the same NaOH dose of 0.2 g/g TS, sludge DD_{COD} improved gradually when sludge TS increased from 2.0 to 11.0%. This is because the molar concentration of NaOH increased proportionally with increased sludge TS when NaOH mass (g/g TS) was kept constant. These results mean that for sludge disintegration using NaOH, sludge thickening can reduce the quantity of alkali required.

When sludge TS increased further from 11.0 to 14.2%, sludge DD_{COD} decreased gradually whether the NaOH dosage was held constant at either 0.1 mol/L or 0.2 g/g TS (Figure 2). In other words, more NaOH was required for the sludge with 11.0–14.2% TS to reach the same disintegration level achieved at a TS of approximately 9.3%. This is because alkaline sludge treatment depends on dissolution or destruction of floc structure and cell walls by the hydroxyl radical (Li et al. 2008). In high-solids sludge, sludge particles are agglomerated and not dispersed adequately, which restricts the effect of the hydroxyl radical and is reflected by increased viscosity. It was found that sludge viscosity increased sharply when solids concentration was higher than 12% (Figure 3). A similar result in the TS range of 9–10% was reported by Ma et al. (2010). Moreover, the released protein and polysaccharides due to alkaline treatment increased sludge viscosity further. High viscosity may hinder sludge homogenization and block mass transfer.

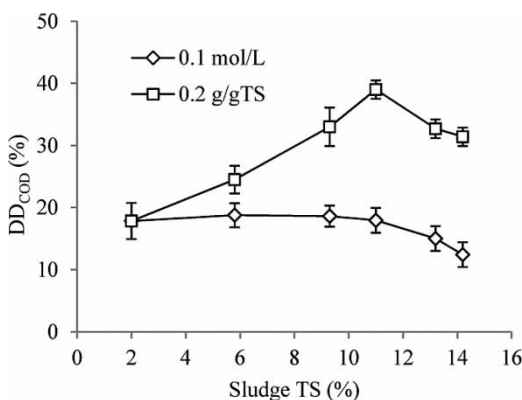


Figure 2 | Variation of sludge DD_{COD} along with sludge TS at NaOH doses of 0.1 mol/L and 0.2 g/g TS.

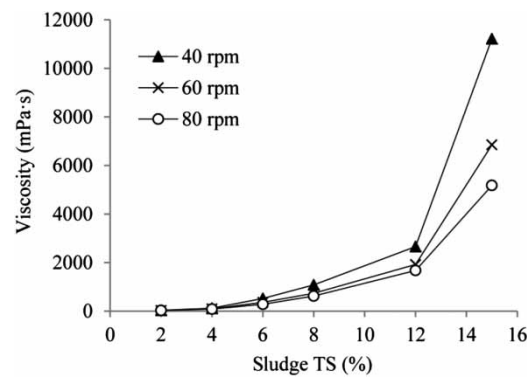


Figure 3 | Sludge viscosities at three spindle speeds in sludge TS ranges of 2–15%.

It was reported that the effective diffusion coefficient decreased drastically when sludge TS increased, with numerical values 56 to 108 times smaller at 8% TS and 11% TS, respectively, than the reference value in water (Bollon et al. 2013). Therefore, the disintegration effect of high-solids sludge was possibly limited by the blocked mass transfer of the hydroxyl radical. It was also noted that DD_{COD} values of high-solids sludge were still higher than those of low-solids sludge at the dose of 0.2 g/g TS, indicating that the negative influence of the high-solids content was partly counteracted by the higher molar concentration of NaOH. Therefore, sludge thickening also can effectively reduce the quantity of alkali required for pretreatment even for the sludge with TS in the range of 11.0–14.2%.

Determination of optimum conditions for alkaline pretreatment

Based on the above results it was possible to construct an empirical model for quantification of sludge disintegration effect due to NaOH treatment. Considering that the value of DD_{COD} changed between 0 and 1, a transmutative power function model was used to describe the disintegration effects of both low-solids sludge and high-solids sludge after 30 min of alkaline treatment

$$DD_{COD} = 1 - \frac{1}{1 + k \cdot C_A^\alpha} \quad (2)$$

where C_A is NaOH dose, mol/L; α is the index of NaOH dose and k is the reaction rate constant related to sludge characteristics and other reaction conditions. For the convenience of calculation, Equation (2) can be changed into Equation (3)

$$\ln \frac{DD_{COD}}{1 - DD_{COD}} = \ln k + \alpha \ln C_A \quad (3)$$

Based on DD_{COD} data shown in Figure 1, k and α can be calculated by linear regression, and the results are shown in Table 2. The significance probability test (p) was used to evaluate the effects of the index on sludge disintegration effects. Using a 5% confidence level ($p < 0.05$), it was observed that NaOH dose showed significant effect on sludge disintegration. The model generated was considered predictive by analysis of variance, and it showed a satisfactory determination coefficient (R^2). This model was used to obtain sludge DD_{COD} at different NaOH doses between studied ranges of sludge TS and to determine the optimum conditions for alkaline pretreatment of high-solids sludge.

It was demonstrated in Figure 2 that sludge DD_{COD} corresponding to a NaOH dose of 0.1 mol/L showed almost no change when sludge TS increased from 2.0 to 11.0% and decreased only slightly when sludge TS increased further from 11.0 to 14.2%. At the other doses, sludge DD_{COD} predicted by Equation (2) showed similar variations in response to increased sludge TS (Figure 4). Therefore, high-solids sludge with TS in the range of 8.0–11.0% is optimum for alkaline treatment from the view of sludge disintegration effect and alkali consumption. Higher solids concentration would significantly increase stirring energy consumption. According to Figure 1, 0.1 and 0.2 mol/L were effective doses for the treatment of low-solids sludge and high-solids sludge, respectively. However, the NaOH dose should be at least lower than 0.15–0.22 mol/L in order to avoid the inhibition effect due to sodium ions (Owen *et al.* 1979). Hence, alkaline pretreatment with 0.05–0.15 mol/L NaOH was tested during AD of high-solids sludge.

BMP tests of high-solids sludge after alkaline pretreatment

BMP of alkali-pretreated sludge was monitored, and the results are shown in Figure 5. During alkaline pretreatment, sludge DD_{COD} was 9.7, 15.8 and 21.7%, respectively, for G1,

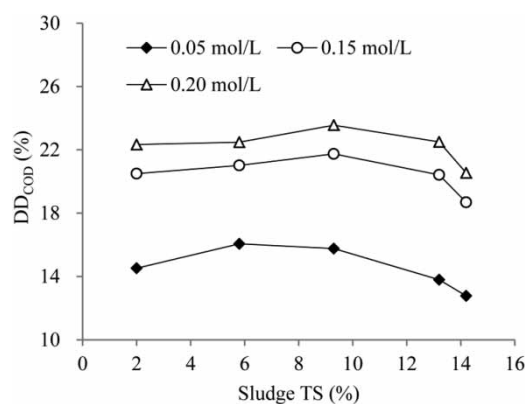


Figure 4 | Variation of sludge DD_{COD} as a function of sludge TS at NaOH doses of 0.05, 0.15 and 0.20 mol/L.

G2 and G3. During the subsequent BMP tests, their specific biogas yields (SBYs) increased from 363 ± 3 mL/g VS_{added} (G0, the control) to 376 ± 2 , 376 ± 5 and 366 ± 1 mL/g VS_{added} for treatments G1, G2 and G3, respectively, with a constant methane proportion of 53–55%. The results indicated that anaerobic bacteria can adapt to the increased salinity environment produced by alkaline pretreatment using NaOH and transform organic substances to biogas continuously. It was noted that these SBYs were close to the value of 383 mL/g VS_{added} , derived from a BMP test with RI/S (the ratio of inoculum VS to substrate VS) of 1.0 (Sri Bala Kameswari *et al.* 2012), and the value of 365 mL/g VS_{added} , obtained from a semi-continuous digester with SRT of 30 days (Dai *et al.* 2013). Therefore, the biogas productivity of high-solids AD was close to that of CAD. Thus, volumetric biogas yields of digesters can increase proportionally with increased TS of feedstock. Hence,

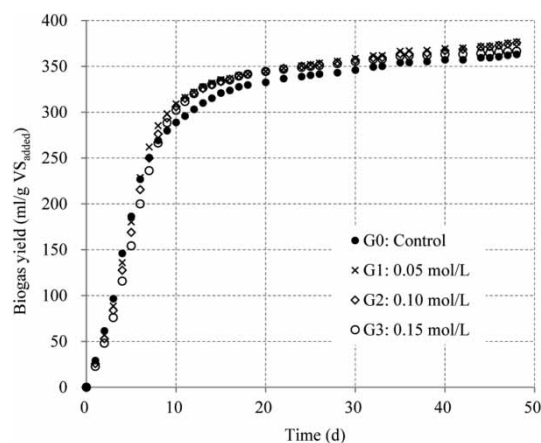


Figure 5 | The effect of NaOH pretreatment on biogas production during the subsequent batch AD.

Table 2 | Model coefficients for alkaline sludge disintegration

Sludge TS (%)	k	α	$p\alpha$	R^2
2	0.53	0.38	0.020	0.87
5.8	0.47	0.30	0.004	0.90
9.3	0.55	0.36	0.004	0.84
13.2	0.58	0.43	0.001	0.94
14.2	0.50	0.41	0.000	0.97

high-solids sludge AD can be operated with smaller digesters than those required for CAD.

Alkaline pretreatment enhanced the performance of high-solids sludge AD, but the increase in accumulative biogas production was limited. This result may be attributed to the characteristics of the sludge used as feedstock in the study, which was composed mainly of primary sludge with a small amount of biofilm sludge. According to its mechanism, alkaline treatment would be expected to have a more obvious effect on excess sludge or waste activated sludge (WAS). For example, alkaline pretreatment enhanced biogas production of WAS by 21% (Navia *et al.* 2002) or 73% (Heo *et al.* 2003). Furthermore, alkaline pretreatment shortened digestion time significantly due to accelerated hydrolysis. After the pretreatment with 0.05 mol/L NaOH (G1), the required digestion time decreased from 48 to 34 days in order to reach the same accumulative biogas yield achieved in G0 (the control treatment with no NaOH) and decreased from 17 to 13 days the time required to reach 90% of the accumulative biogas yield achieved in G0.

Alkaline pretreatment also resulted in a lag phase at the beginning of the BMP tests due to Na⁺ inhibition. After the lag phase, the biogas production of the reactors with alkaline pretreatment still exceeded that of the control. The lag phases were 5, 6 and 8 days, respectively, for G1, G2 and G3, which had progressively higher Na⁺ concentrations in the digestate. Aside from this, high concentration of Na⁺ also reduced biogas yield. Therefore, the performances of G1 and G2 were better than that of G3. Comparing G1 with G2, more organic substances were dissolved in G2 due to a higher NaOH dose. Although some organic substances were not dissolved directly due to alkaline pretreatment in G1, sludge floc structure and cell wall still became loose or disrupted, and the undissolved organic substances were exposed. These substances were easily solubilized and utilized during the subsequent AD. Therefore, G1 exhibited a similar performance to G2. From the view of biogas production and NaOH consumption, 0.05 mol/L NaOH was the optimum dose for sludge pretreatment. This dose is in accordance with the range of 0.04–0.1 mol/L shown to be optimum for the treatment of low-solids sludge (Li *et al.* 2012).

CONCLUSIONS

Alkaline treatment effectively disintegrated both low-solids sludge and high-solids sludge. At NaOH doses lower than

0.2 mol/L, the disintegration level of low-solids sludge was almost uninfluenced by sludge concentration. Although a higher molar concentration of NaOH was required for sludge with 11.0–14.2% TS to reach the same disintegration level as for low-solids sludge, the required mass of NaOH still decreased significantly due to sludge thickening. The relation between sludge DD_{COD} and NaOH dose can be described by a transmutative power function model. Alkaline pretreatment with 0.05 mol/L NaOH for 30 min was the optimal condition for high-solids sludge AD. The pretreatment can avoid Na⁺ inhibition of anaerobic bacteria and effectively accelerate the subsequent AD.

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